



U.S. ARMY DUGWAY PROVING GROUND
HAZARDOUS WASTE STORAGE PERMIT

APPENDIX A

QUALITY ASSURANCE PROGRAM PLAN FOR THE ANALYSIS OF CHEMICAL AGENT-RELATED WASTE

Issued: December 1997

Revised: April 2001

1.0 INTRODUCTION

The U.S. Army Dugway Proving Ground (DPG) originally submitted this Quality Assurance Program Plan (QAPP) in accordance with State of Utah (State) Solid and Hazardous Waste Control Board Consent Order (CO) Number 9505024. This CO required DPG to prepare and submit a QAPP prepared in accordance with the U.S. Environmental Protection Agency (EPA) Interim Guidelines for Preparing Quality Assurance Project Plans, EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), and other appropriate and relevant guidance documents. Upon Utah Division of Solid and Hazardous Waste approval, appropriate QAPP information has been incorporated into the Waste Analysis Plan (WAP) of DPG's hazardous waste storage permit (Permit).

The DPG Directorate of Environmental Programs (DEP) and Chemical Test Division (CTD) have jointly prepared this QAPP for DPG's chemical agent-related waste analysis program. This QAPP outlines the policies, requirements, procedures, and responsibilities established to support analysis of chemical agent-related wastes conducted at DPG. This document also provides specific quality assurance (QA) and quality control (QC) procedures necessary to generate data of acceptable quality and completeness.

The purpose of this section is to outline the QAPP policies regarding collection and analysis of chemical agent-related wastes conducted on-site at DPG. Section 1.1 discusses the purpose, as well as the outline and documentation sources, for the QAPP. Sections 1.2 and 1.3 contain DPG's QAPP quality and ethics policies, respectively.

1.1 PURPOSE

The purpose of the QAPP is to ensure the quality and defensibility of chemical agent-related analytical data. As used in this QAPP, chemical agents include the following compounds:

- ◆ GA - Ethyl N,N-dimethylphosphoramidocyanidate - Tabun
- ◆ GB - Isopropyl Methylphosphonofluoridate - Sarin
- ◆ GD - Pinacolyl Methylphosphonofluoridate - Soman
- ◆ GF - Cyclohexyl Methylphosphonofluoridate
- ◆ HD - Bis-2-chloroethylsulfide - Distilled Mustard
- ◆ HN-3 - tris-2-chloroethylamine - Nitrogen Mustard
- ◆ VX - O-ethyl-S-[2-diisopropylaminoethyl] methylphosphonothiolate
- ◆ Lewisite - Dichloro-(2-chlorovinyl) arsine

The quality systems described in this QAPP have been developed to comply with local and national standards for environmental laboratories producing data for Resource Conservation and Recovery Act (RCRA) compliance. Guidance in the preparation of this QAPP was obtained from the following sources:

- ◆ "Interim Guidelines and Specifications for Preparing Quality Assurance Program Plans" (EPA, 1983)

- ◆ “Preparation Aids for the Development of Category 1 Quality Assurance Program Plans,” (EPA, 1991)
- ◆ “Interim Draft Requirements for Preparing Quality Assurance Project Plans,” (EPA, 1984)
- ◆ “Test Methods for Evaluating Solid Waste, Update III,” SW-846 (EPA, 1997)

This QAPP contains 13 sections. Section 1.0 is an introduction to the QAPP. Section 2.0 describes the project organization and details the responsibilities of key project personnel. Section 3.0 outlines required personnel qualifications and personnel training. Section 4.0 describes the facilities, equipment, and supplies used to generate chemical agent-related waste data. Section 5.0 describes the required format, development, approval, and control of methods and other documents related to the QAPP. Section 6.0 outlines the documentation and procedural requirements for sample collection. Section 7.0 describes several general laboratory procedures including sample receiving, sample handling, and labware cleaning. Section 8.0 discusses the calibration requirements for laboratory and field instrumentation. Section 9.0 outlines the project data quality objectives (DQOs), analytical method performance, method detection limits, and reporting limits. Section 10.0 describes analytical data management including recording, reduction, reporting, review, and validation. Section 11.0 identifies the methods for laboratory quality assessment including control charts, proficiency test samples, audits, and reviews. Section 12.0 outlines the requirements for implementing and documenting corrective action procedures. Section 13.0 defines the terms used in the QAPP.

1.2 QUALITY POLICY

DPG is committed to producing high quality analytical data that is technically and legally defensible. As part of DPG’s commitment to high quality data, project management will ensure that employees and contractors have sufficient experience and training to perform QAPP-related duties and procedures. Sample collection, sample handling, instrument calibration, sample analysis, and related activities will be conducted and documented as described in this QAPP and related methods. Routine QA samples will be prepared, analyzed, and reviewed according to method-specific procedures and at specified frequencies. Regular internal and external audits will be conducted and documented to assess compliance with the QAPP and methods. Corrective action will be initiated and completed to address discrepancies or problems noted at any point in the process.

1.3 ETHICS AND CONFIDENTIALITY POLICY

Without exception, DPG requires honest and ethical behavior of its employees and contractors. Employees and contractors are required to fully and accurately represent all aspects of their QAPP-related activities. Personnel must never intentionally report dates, data, or times other than those actually observed. Personnel must never intentionally represent another individual’s activities as his/her own or misrepresent any other aspect of the analytical process. Alterations, additions, and/or deletions to data, reports, and other documentation must be made according to scientifically acceptable standards as described in this QAPP. Employees and contractors are required to inform, in a timely manner, DPG or project management of any such unethical behavior observed of other employees.

In a similar manner, employees and contractors are required to protect the integrity and confidentiality of sample and data information. Except as permitted in writing, data are released only to the submitting party. Care should also be taken when transmitting data by facsimile or other electronic means. Sample information, results of analyses, and other proprietary and/or sensitive information must not be discussed with, or transmitted to, individuals outside DPG without DPG's authorization. Environmental, safety, or other concerns should be communicated within the chain of command at DPG. Likewise, auditors and other individuals visiting the facility are required to maintain the confidentiality of proprietary and/or sensitive information.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

This section describes the requirements and responsibilities of specific personnel involved with the sampling and analysis of chemical agent-related waste. Section 2.1 describes the DPG and QAPP organizations. Section 2.2 outlines the QAPP-related responsibilities of key project personnel.

2.1 DPG ORGANIZATION

Two DPG organizations are jointly responsible for the establishment and implementation of the QAPP. They include the DEP and the West Desert Test Center (WDTC) as described below in Figure 2.1.

2.1.1 DEP Organization

DEP personnel coordinate and manage the environmental conservation, restoration and compliance projects at DPG. The DEP Compliance and Restoration Division (CRD) oversees RCRA, Installation Restoration Program (IRP), Underground Storage Tank (UST), and other compliance-related projects. The DEP RCRA Coordinator works directly with the State of Utah Division of Solid and Hazardous Waste (DSHW) to ensure compliance with applicable RCRA permits and regulations.

2.1.2 WDTC Organization

The WDTC provides analytical testing in support of DPG's chemical agent defense programs. The WDTC CTD includes project management and analytical support personnel housed in the Combined Chemical Test Facility (CCTF). The CTD Analytical Branch provides routine and specialized analytical support including environmental testing. Analytical Branch personnel and contractors are responsible for sample collection and analytical support activities described in this QAPP.

2.2 PERSONNEL RESPONSIBILITIES

The individuals listed below are responsible for conducting the various activities detailed in this QAPP as well as implementing the methods and operating procedures listed in Section 5.0

2.2.1 Compliance and Restoration Division Chief

The DEP CRD Chief has the following responsibilities relative to the analysis of chemical agent-related waste:

- ◆ Provide the RCRA Coordinator with the necessary authority, personnel, equipment and facilities to properly manage the waste sampling and analysis activities as described in this QAPP
- ◆ Promptly review and respond to the QC deficiencies reported by the DEP RCRA Coordinator

2.2.2 Chemical Test Division Chief

The WDTC CTD Chief has the following responsibilities relative to the analysis of chemical agent-related waste:

- ◆ Provide overall management of CTD activities including the sampling and analysis of chemical agent-related waste samples
- ◆ Provide the Analytical Branch Chief with the necessary authority, personnel, equipment and facilities to properly manage the waste analysis activities as described in this QAPP
- ◆ Promptly review and respond to QC deficiencies reported by DEP management, the Analytical Branch Chief, and/or the Analytical Branch QC Chemist

2.2.3 Compliance and Restoration Division Resource Conservation and Recovery Act Coordinator

The RCRA Coordinator has the following responsibilities relative to the analysis of chemical agent-related waste:

- ◆ Read, understand, and oversee the compliance activities described in this QAPP and the QAPP-related methods
- ◆ Act as the central point of contact and authority for RCRA permitting activities associated with this QAPP
- ◆ Facilitate DPG compliance with applicable Federal and State of Utah RCRA laws and regulations and U.S. Army waste management regulations
- ◆ Ensure that data is independently validated

2.2.4 Chemical Test Division Analytical Branch Chief

The Analytical Branch Chief has the following responsibilities relative to the analysis of chemical agent-related waste:

- ◆ Read, understand and direct the sampling, analysis, documentation, and QC activities described in this QAPP and the QAPP-related methods
- ◆ Ensure that all data reported by the laboratory is of high quality as well as technically and legally defensible
- ◆ Ensure that technical and support personnel are have sufficient qualifications and training to perform their assigned functions
- ◆ Promptly review and respond to QC deficiencies and complaints reported by the QC Chemist, the CRD RCRA Coordinator, and/or other clients

2.2.5 Chemical Test Division Quality Control Chemist

The QC Chemist has the following responsibilities relative to the analysis of chemical agent-related waste:

- ◆ Read, understand and assess the QC activities described in this QAPP and the related methods

- ◆ Review analytical data and reports to ensure compliance with this QAPP and the QAPP-related methods
- ◆ Conduct an semiannual internal audit of sampling and analysis activities to ensure compliance with this QAPP and the QAPP-related methods
- ◆ Conduct an annual review of this QAPP and the QAPP-related methods
- ◆ Ensure performance of annual precision and accuracy studies for QAPP-related methods and analytes
- ◆ Maintain records of ongoing personnel training for QAPP-related activities
- ◆ Maintain a corrective action program to review and respond to QC deficiencies and complaints

2.2.6 Sample Collection Personnel

Sample collection personnel have the following responsibilities relative to the analysis of chemical agent-related waste:

- ◆ Read, understand and follow the QC guidelines as described in this QAPP
- ◆ Read, understand and follow sample collection procedures as described in the QAPP-related sampling methods
- ◆ Accurately and honestly record pertinent information and complete required documentation as described in the QAPP-related sampling methods
- ◆ Promptly deliver samples to the CCTF for analysis

2.2.7 Analytical Personnel

Analytical personnel include DPG and subcontractor chemists and technicians located in the CCTF, as well as Miniature Continuous Air Monitoring System (MINICAMS[®]) operators and other field analytical personnel. They have the following responsibilities relative to the analysis of chemical agent-related waste:

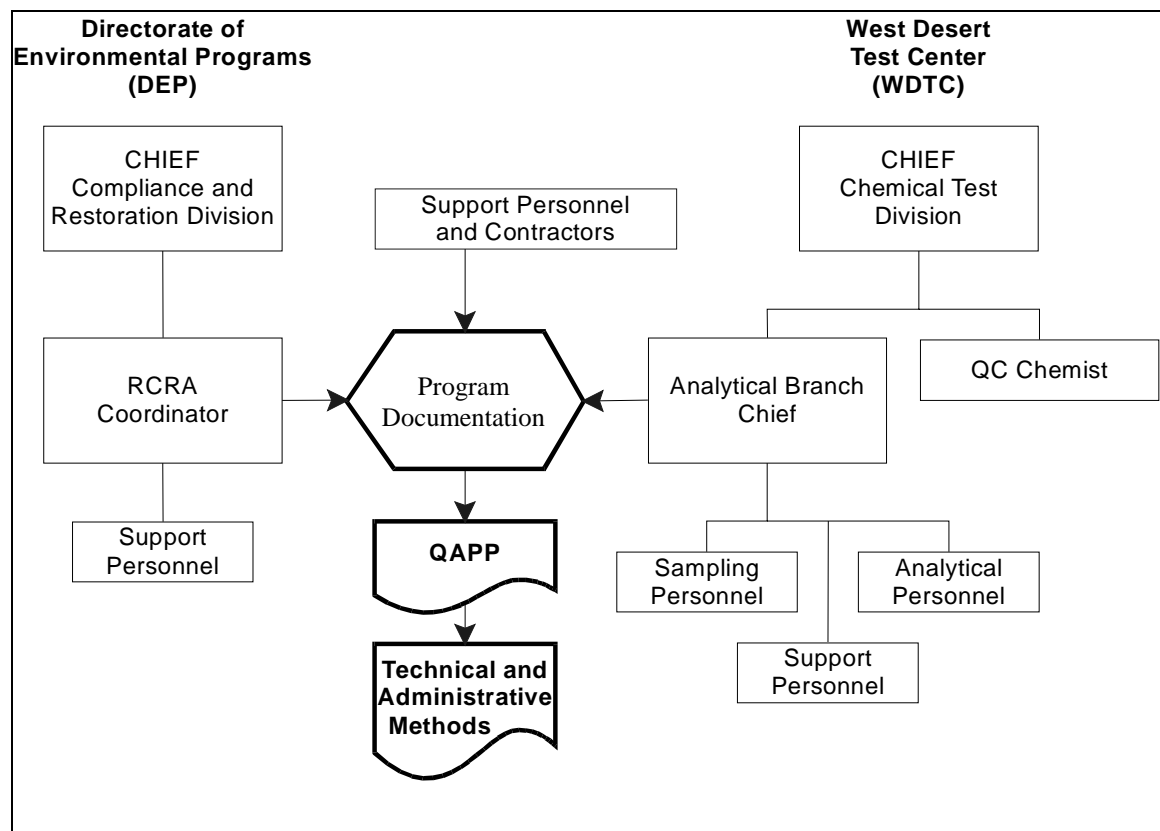
- ◆ Read, understand and follow the QC guidelines as described in this QAPP
- ◆ Read, understand and follow the procedures as described in the QAPP-related analytical and administrative methods
- ◆ Accurately and honestly record pertinent information and complete required documentation as described in the QAPP-related analytical and administrative methods
- ◆ Ensure that analytical results are accurate, technically defensible, and meet the QC requirements as described in this QAPP and the QAPP-related analytical and administrative methods
- ◆ Complete ongoing training as described in Section 3.0
- ◆ Demonstrate training effectiveness by successful completion of method-required quality control such as blanks, calibration verification standards, spikes, and spike duplicates
- ◆ Maintain data quality and client confidentiality for all chemical agent-related results by following the reporting procedures as described in this QAPP and the QAPP-related analytical and administrative methods
- ◆ Properly operate and regularly maintain laboratory analytical instrumentation and equipment
- ◆ Report technical and quality problems immediately to the QC Chemist or Analytical Branch Chief

2.2.8 Laboratory Support Personnel

Laboratory support personnel include sample custodians, documentation clerks, data package assembly personnel, and others. They have the following responsibilities relative to the analysis of chemical agent-related waste:

- ◆ Read, understand and follow the QC guidelines as described in this QAPP
- ◆ Read, understand and follow the pertinent analytical and administrative procedures as described in the QAPP-related methods
- ◆ Accurately and honestly record pertinent information and complete required documentation as described in the QAPP-related methods
- ◆ Receive samples for analysis as described in the QAPP-related methods
- ◆ Verify field and custody documentation, preservation and holding times as described in the QAPP-related methods
- ◆ Collect and maintain sampling and analytical records as described in the QAPP-related methods
- ◆ Prepare data packages for external validation as described in the QAPP-related methods
- ◆ Maintain a document control system for this QAPP and the QAPP-related methods
- ◆ Report technical and quality problems immediately to the QC Chemist or Analytical Branch Chief

Figure 2-1. QAPP Organization Chart.



QAPP	Quality Assurance Program Plan
QC	Quality Control
RCRA	Resource Conservation and Recovery Act

3.0 PERSONNEL QUALIFICATIONS AND TRAINING

DPG recognizes that well trained and experienced personnel are the laboratory's most important resource. All personnel contributing to the quality of chemical agent-related waste data must have an adequate combination of education and experience to perform their required functions. Individuals who work from the guidance of this QAPP must be familiar with the general QAPP requirements as well as the applicable specific requirements detailed in the QAPP-related methods and procedures. Ongoing training and proficiency demonstration is also required of all personnel who implement the requirements of this QAPP.

This section outlines the requirements for personnel qualifications and training. Section 3.1 describes the technical qualifications and experience required of key QAPP personnel. Requirements for continuing education and training are outlined in Section 3.2.

3.1 PERSONNEL QUALIFICATIONS

The Analytical Branch Chief is ultimately responsible for the quality and defensibility of analytical data produced in the laboratory. Each person contributing to the quality of laboratory data must have sufficient education, knowledge, and training to properly perform their QAPP-related functions.

The administrative and documentation requirements of environmental analyses are often different from those of the military programs supported by the Analytical Branch laboratory. For this reason, specific environmental laboratory experience is indispensable for key environmental testing and management personnel. In addition to specific requirements set forth by DPG management, key environmental laboratory personnel should have the following minimum qualifications:

3.1.1 Chemical Test Division Analytical Branch Chief

The Analytical Branch Chief (or a designee overseeing the QAPP-related analyses) should have a minimum of a bachelor's degree in the chemical, environmental or biological sciences, with a minimum of 24 college semester credit hours in chemistry. This person should have at least two years of experience performing and at least two years supervising the analysis of hazardous waste and other environmental samples.

3.1.2 Chemical Test Division Quality Control Chemist

The QC Chemist should have a minimum of a bachelor's degree in science or engineering, with a minimum of 24 college semester credit hours in chemistry. This person should also have formal training in general statistics and demonstrate a working knowledge of environmental QC methods and procedures.

3.1.3 Analytical Chemists

Analytical Chemists performing environmental analyses should have a minimum of a bachelor's degree in the chemical, environmental, or biological sciences, with a minimum of 24 college semester credit hours in chemistry. In addition to the DPG-required experience analyzing chemical agents, analytical chemists should demonstrate a working knowledge of environmental QC methods and procedures.

3.1.4 Other Technical Personnel

Where possible, sampling, analytical, and other technical personnel should have formal training in their area(s) of responsibility. Such training could come from in-house or outside sources.

3.2 PERSONNEL TRAINING

The training program will include initial and annual QAPP training, method-specific training, and other training as described below. Training documentation will be maintained accessible and up-to-date by the supervising organization. Pre-employment information for DPG employees is maintained by the DPG Civilian Personnel Office (CPO).

3.2.1 Initial and Annual QAPP Training

Employees and subcontractors involved in the collection, handling, analysis, and/or processing of chemical agent-related wastes will undergo initial QAPP training. This training will familiarize personnel with QAPP quality and ethics policies, analytical and administrative methods, documentation requirements, and other information contained in this QAPP and related methods. An understanding of the information contained in this QAPP will be demonstrated by successful completion of a written examination developed by the DEP RCRA Coordinator.

QAPP personnel are required to participate in refresher training on an annual or more frequent basis. Annual QAPP training will include a review of general QAPP concepts, methods status, and regulatory changes. An understanding of the information contained in this QAPP will be demonstrated by successful completion of a written examination developed by the DEP RCRA Coordinator.

3.2.2 Method Proficiency Demonstration

In addition to possessing sufficient qualifications and experience as outlined in Section 3.1, personnel performing QAPP-related methods and procedures must demonstrate annual method-specific proficiency. In order to demonstrate proficiency for an analytical method or procedure, personnel must read and understand the method, perform the method under the direction of a qualified supervisor or mentor, and demonstrate the ability to consistently perform the activity within method required specifications. In order to demonstrate proficiency for an administrative method, personnel must document that they have read and understand the method. The supervisor or mentor may include a written test, blind audit samples, or other activities as part of the initial and/or ongoing proficiency assessment. Successful demonstration of method

proficiency is approved by the supervisor or mentor and is documented in the training records (Exhibit 3-1).

3.2.3 Other Training

The DEP RCRA Coordinator encourages ongoing training and continuous improvement for QAPP personnel. Where necessary, formal instruction should be sought from outside sources, such as for instrumentation and/or software operation. Other sources of continuing instruction and education include in-house seminars and training sessions, technical subscriptions, and participation in professional organizations.

3.2.4 Training Documentation

A training file will be maintained for QAPP personnel. QAPP training files will include documentation of required pre-employment education and experience (such as a resume), copies of relevant certificates and degrees, and other qualification information. Method Training Forms, proficiency sample results, written test results, and relevant on-the-job training will also be documented in the employee's training file. The QA Chemist will ensure that applicable Analytical Branch training information is maintained and available for inspection. The DPG Civilian Personnel Office maintains pre-employment information for DPG employees.

Exhibit 3-1. Example of QAPP Method Training Record

DPG QAPP PERSONNEL METHOD TRAINING RECORD				
Employee Name:			Training Period:	
Supervisor:				
✓ Required	Method Number	Method Title	Date Completed	Approval
ADMINISTRATIVE METHODS				
	ADM-008R	Recording, Revising, and Issuing Controlled Documents		
	ADM-011R	Data Package Preparation		
	ADM-013R	Laboratory Sample Receipt and Login		
	ADM-014R	Format for Regulatory Analytical Methods		
	ADM-015R	Training Requirements and Records		
	ADM-016R	Format for Regulatory Administrative Methods		
	ADM-017R	Recording Analytical Data		
	ADM-018R	Maintaining Custody for Regulated Waste Samples		
	ADM-019R	Quality Control Review of Analytical Data		
	ADM-021R	Reporting Analytical Data		
	ADM-022R	Peer Review of Analytical Data		
SAMPLING METHODS				
	CL-022R	Sampling Solid Wastes with DAAMS		
	CL-055R	Sampling Liquid Wastes		
	CL-056R	Sampling Solid Wastes with Bubblers		
	CL-057R	Sampling Soils		
ANALYTICAL METHODS				
	CL-001R	Chemical Agents in Liquid Wastes by Gas Chromatography		
	CL-002R	Chemical Agents in Liquid and Solid Wastes by GC/MS		
	CL-004R	Chemical Agents in Soils by Gas Chromatography		
	CL-025R	Lewisite in Bubblers, Liquids, and Soils by Gas Chromatography		
	CL-027R	Chemical Agents in Bubblers by Enzymatic Assay - Technicon Method		
	CL-044R	Chemical Agent Monitoring using Field MINICAMS®		
	CL-045R	Lewisite Monitoring using Field MINICAMS®		
	CL-052R	Chemical Agents in DAAMS by Gas Chromatography		
OTHER METHODS				
	RCRA	RCRA Hazardous Waste Management		
	WAP/QAPP	RCRA Waste Analysis Plan and QA Program Plan		
	CL-021R	General Labware Washing and Maintenance		
	CL-069R	Precision Labware Washing and Maintenance		
	ADM-020R	Planning Sampling and Analysis Operations		
	ADM-012R	Validation of Analytical Data		

4.0 FACILITIES AND EQUIPMENT

Buildings 4165 and 4156 in the Ditto Technical Center are often referred to as CCTF. Figure 4-1 shows the layout of the CCTF. This modern 35,000-square-foot facility supports the testing of protective clothing and masks, detectors, and decontamination systems using chemical agents and simulants as challenge materials. Testers determine agents, simulants, and other analytes in samples which were collected in laboratory and chamber trials. In addition, the facility supports the analysis of environmental samples from DPG operations to ensure compliance with Federal, State, and local regulations.

4.1 LABORATORY FACILITIES

The CCTF laboratory (Building 4156) has been specifically designed for the analysis of chemical agents. The laboratory is designed to provide a safe and comfortable working environment. Separate work areas are provided for labware cleaning, sample storage, sample preparation, sample analysis, sample disposal, records retention, and other laboratory activities. Offices are located around the perimeter of the building to provide easy access.

The CCTF (Building 4156) provides a high degree of environmental control. Variables such as temperature, humidity, ventilation, and lighting can be controlled and monitored as necessary. Environmental conditions that are specified in a particular method are monitored and documented.

The laboratory is environment-friendly. A double-wall drain system and a 5,000-gallon holding tank contain contaminated water from any agent spill cleanup or from emergency shower use. The exhaust air from all laboratory areas (not only fume hoods) is charcoal-filtered before it is returned to the atmosphere. In addition, a demand-controlled variable-volume ventilation system minimizes the volume of air requiring heating or cooling. Heat-recovery coils downstream from the fume hood filter units recover energy from exhausted air. Motion sensors automatically turn off lights in unoccupied rooms. Thick wall and roof insulation and heat-reflector windows minimize heat transfer through the building shell.

4.2 LABORATORY EQUIPMENT

The CCTF is equipped to safely test, analyze, and process chemical agent-related wastes. Laboratory analytical capabilities include:

- ◆ Atomic absorption spectroscopy (inorganic substances, heavy metals, environmental contaminants)
- ◆ Automated thermal desorption chromatography systems (analysis of organic compounds)
- ◆ Continuous vapor monitoring (MINICAMS[®] which employ gas chromatography (GC) for low-level vapor; infrared spectroscopy for high-level vapor)
- ◆ GC (flame ionization, flame photometric, photo ionization and thermal conductivity detectors)
- ◆ GC/mass spectrometry (MS)

- ◆ High-performance liquid chromatography
- ◆ Infrared spectrometry
- ◆ Spectrofluorometry
- ◆ UV-visible spectrophotometry.

Formal training and experience is required to operate most analytical equipment (see Section 3.2.3). Manuals and instructions for the operation of test equipment are maintained up-to-date in the laboratory.

4.3 EQUIPMENT MAINTENANCE

Routine equipment and instrument maintenance minimizes down time and prevents unexpected problems within the laboratory. Routine maintenance is performed on all laboratory instrumentation according to the manufacturer's recommendations. Where possible, manufacturer service contracts are maintained on major pieces of equipment. The DPG Calibrations Unit performs calibration of measurement equipment, such as balances and flow meters, at least annually. Routine maintenance for MINICAMS® is performed at least annually by factory trained service personnel.

Maintenance of all major laboratory and field instrumentation is recorded in instrument maintenance logbooks. Maintenance logbooks document all routine (change column, pump tubing, etc.) and non-routine (troubleshooting, instrument service, etc.) maintenance operations.

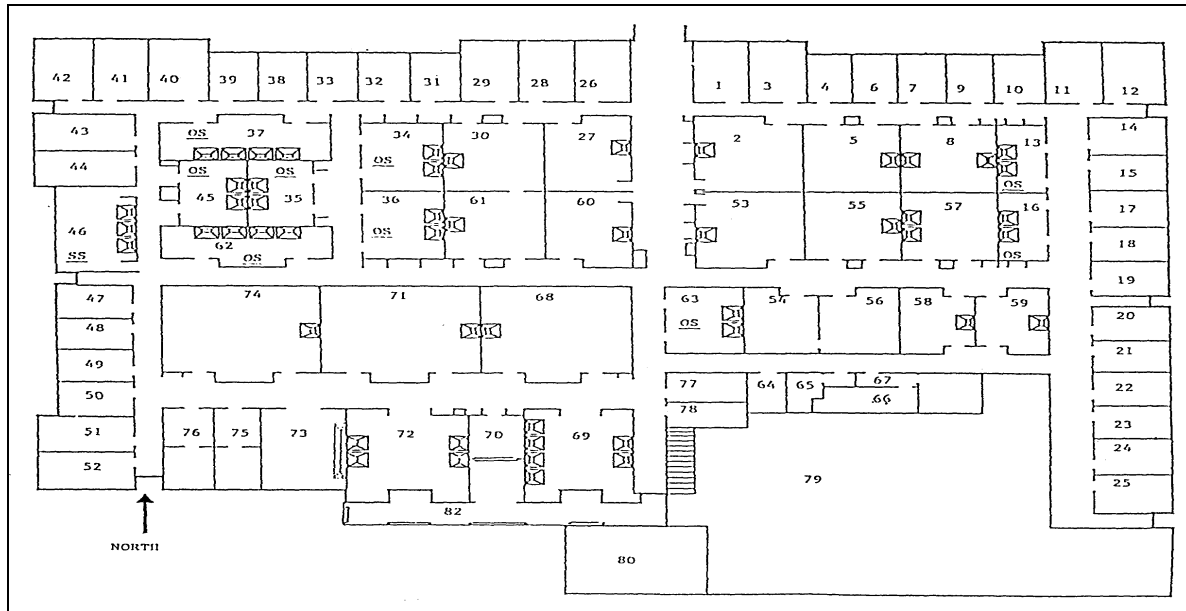
4.4 SAFETY

A sophisticated exhaust system, with redundant fans, controls, and alarms, provides the airflow in fume hoods used for all agent operations. The building's pressurization system keeps laboratory rooms at a lower air pressure than corridors, which in turn are kept at a lower pressure than the offices. Emergency generator capacity supports the fume hoods, ventilation system, egress lighting, and other essential equipment in the event of a power loss. Emergency showers and eyewashes are provided in the laboratories and corridors. Epoxy and stainless steel work surfaces and interior finishes are resistant to chemical agents. A piping system for commonly used compressed gases reduces the need to store gas cylinders in the laboratory rooms. A separate bulk solvent storage building minimizes storage of flammable materials in the laboratory building.

4.5 SECURITY

A security fence surrounds the CCTF, restricting access to the facilities. Access to the CCTF is controlled by a security gate through which only authorized personnel or escorted visitors are allowed. During certain chemical agent-test operations, a security guard is posted at the fence and only authorized personnel are allowed to enter the area. All agent storage areas have concrete vault construction, high-security hardware and locks, and an intrusion detection system. Archived laboratory data is stored in a locked room equipped with fire suppression capability.

Figure 4-1. Floorplan of Building 4156, Combined Chemical Test Facility.



5.0 OPERATING PROCEDURES AND DOCUMENT CONTROL

The development and routine use of written operating procedures promotes consistency and reproducibility within the laboratory. Activities related to the sampling, analysis, and reporting of chemical agent-related wastes are documented in WDTC administrative (designated “ADM-XXX”) and analytical (designated “CL-XXX”) methods. Table 5-1 lists the sampling, analytical and administrative methods associated with this QAPP.

This section describes the format for administrative and analytical methods, analytical method development and approval, and document control for methods referenced in this QAPP. Sections 5.1 and 5.2 outline the requirements for administrative and analytical methods. Analytical method development is described in Section 5.3 and document control is described in Section 5.4.

5.1 FORMAT FOR ADMINISTRATIVE METHODS

Administrative methods detail the requirements for QAPP-related administrative activities such as sample receiving, laboratory documentation, data review, data reporting, and personnel training. Preparing administrative methods is described in WD-C METHOD ADM-016R: “Format for Regulatory Administrative Methods.” Each administrative method will include, where applicable, the following elements:

- ◆ Title / Approval Page
- ◆ Header (method number, title, revision number, etc.)
- ◆ Scope and Application
- ◆ Terminology
- ◆ Procedure
- ◆ References
- ◆ Figures, Tables, and Exhibits

5.2 FORMAT FOR ANALYTICAL METHODS

Analytical methods detail the requirements for QAPP-related analytical activities such as sample collection, sample analysis, and labware washing. Preparing analytical methods is described in WD-C METHOD ADM-014R: “Format for Regulatory Analytical Methods.” Each analytical method will include, where applicable, the following elements:

- ◆ Title / Approval Page
- ◆ Header (method number, title, revision number, etc.)
- ◆ Scope and Application
- ◆ Scientific Basis
- ◆ Terminology
- ◆ Safety
- ◆ Apparatus and Reagents
- ◆ Standards and Quality Control
- ◆ Procedure

- ◆ Data Reduction and Assessment
- ◆ References
- ◆ Figures, Tables, and Exhibits

5.3 ANALYTICAL METHOD DEVELOPMENT AND APPROVAL

Methods approved for characterizing chemical agent-related wastes are listed in the Waste Analysis Plan (Appendix 1). Where it is necessary to change existing methods or employ new methods for these analyses, the methods or changes will be subject to agreement between the laboratory, the DPG RCRA Coordinator, and the State of Utah DSHW. New or updated methods must be fully documented and approved before they are implemented. Exceptional departures from approved methods and procedures must be clearly documented and approved by the RCRA Coordinator and the Utah DSHW.

During method development, the laboratory must demonstrate that the analytes of interest can be determined in the expected matrices, and that precision, accuracy, and detection limits are adequate for the intended use of the data. Factors to be considered during method development include:

- ◆ Sampling and preservation requirements
- ◆ Stability of samples
- ◆ Extraction efficiencies
- ◆ Stability of extracts
- ◆ Analytical matrix effects and interferences
- ◆ Method detection limits
- ◆ Reporting limits
- ◆ Precision
- ◆ Accuracy

5.4 DOCUMENT CONTROL

Key documents within the laboratory, such as this QAPP and associated methods, are controlled to ensure that changes are made in a uniform manner and that only the latest revision of each document is being used. The Dugway Document Control Clerk maintains original QAPP documents and distributes controlled copies to designated personnel. Controlled documents may be electronic or hardcopy. Controlled hardcopy documents are sequentially numbered and designated with a controlled document stamp (See Exhibit 5-1).

Technical or administrative personnel may initiate revisions to controlled documents. Revisions to QAPP-related documents must be approved by DEP. The State of Utah DSHW must also approve significant changes to sampling or analytical methods.

Table 5-1. Administrative, Sampling, and Analysis Methods for Chemical Agent-Related Wastes.

Method Number	Method Title
ADMINISTRATIVE METHODS	
ADM-008R	Recording, Revising, and Issuing Controlled Documents
ADM-011R	Data Package Preparation
ADM-012R	Validation of Analytical Data
ADM-013R	Laboratory Sample Receipt and Login
ADM-014R	Format for Regulatory Analytical Methods
ADM-015R	Training Requirements and Records
ADM-016R	Format for Regulatory Administrative Methods
ADM-017R	Recording Analytical Data
ADM-018R	Maintaining Custody for Regulated Waste Samples
ADM-019R	Quality Control Review of Analytical Data
ADM-020R	Planning Sampling and Analysis Operations
ADM-021R	Reporting Analytical Data
ADM-022R	Peer Review of Analytical Data
SAMPLING METHODS	
CL-022R	Sampling Solid Wastes with DAAMS
CL-055R	Sampling Liquid Wastes
CL-056R	Sampling Solid Wastes with Bubblers
CL-057R	Sampling Soils
ANALYTICAL METHODS	
CL-002R*	Chemical Agents in Liquid and Solid Wastes by GC/MS
CL-025R	Lewisite in Bubblers, Liquids, and Soils by Gas Chromatography
CL-027R	Chemical Agents in Bubblers by Enzymatic Assay - Technicon Method
CL-044R	Chemical Agent Monitoring using Field MINICAMS®
CL-045R	Lewisite Monitoring using Field MINICAMS®
CL-052R	Chemical Agents in DAAMS by Gas Chromatography
OTHER METHODS	
CL-021R	General Labware Washing and Maintenance
CL-069R	Precision Labware Washing and Maintenance
DP-0000-M-73	Preparation of Standard Solutions

*CL-002R replaces analytical methods CL-001R and CL-004R

Exhibit 5-1. Example of Controlled Document Stamp.



(Note: Red ink is normally used)

6.0 SAMPLE COLLECTION

Proper sample collection is critical to making correct waste disposal and treatment decisions. Sample collection personnel must ensure that samples delivered to the laboratory are representative of the waste in question. Sample collection, preservation, and transportation procedures must minimize sample loss and analyte degradation. Additionally, sample collection personnel must ensure samples and QC samples (blanks, duplicates) are collected in sufficient volume for laboratory analysis.

General sample collection protocols, equipment, preservation, storage, and QA/QC procedures are described below. Sections 6.1 and 6.2 describe the requirements for generator and sample collection planning and documentation. Section 6.3 describes the cleaning of sample collection equipment and containers. Sections 6.4 through 6.6 describe collecting liquid, soil, and solid samples. Section 6.7 describes collecting field QC samples. Sections 6.8 and 6.9 describe the requirements for maintaining sample custody and requesting sample analysis. Communicating potential safety concerns and delivering samples to the laboratory are described in Sections 6.10 and 6.11.

6.1 PLANNING AND DOCUMENTING WASTE GENERATION ACTIVITIES

In order to minimize unnecessary sample collection and analysis, waste generation activities should be well planned and documented. Planning of chemical agent related waste handling activities is detailed in WD-C METHOD: ADM-020R: "Planning Sampling and Analysis Operations." For chemical agent-related wastes, the waste generator (such as the Test Officer or building operator) is responsible for clearly documenting and communicating the history of the samples to be tested.

6.2 DOCUMENTING SAMPLE COLLECTION ACTIVITIES

Pertinent sample collection information is recorded as it occurs. The following information should be recorded in the field logbook or worksheet, as applicable:

- ◆ Name(s) of sample collection personnel
- ◆ Collection date
- ◆ Collection time for each sample
- ◆ Start time, end time, and flow rate for air samples
- ◆ Location of sample collection
- ◆ Type of waste (liquid decontamination solution, solid test item, etc.)
- ◆ Sample identification (drum number, barcode number, etc.)
- ◆ Description of sample (color, consistency, tentative identification, historical information, etc.)
- ◆ Number of phases present and description of each phase
- ◆ Identifying marks or number on container
- ◆ Sample collection equipment, method and description
- ◆ Personal protective equipment used
- ◆ Environmental conditions (temperature, moisture, etc.)

- ◆ Unusual or hazardous conditions
- ◆ Other observations

All sample containers (or sample collection devices such as bubblers and sorbent tubes) must be clearly marked to avoid misidentification. Affix tags or self-adhesive labels to the sample containers before, or at the time of, sample collection. Sample labels (or accompanying paperwork if samples are small) should include the following information, as applicable:

- ◆ Unique field sample identification number
- ◆ Name of collector
- ◆ Date of collection
- ◆ Time of collection
- ◆ Start time, end time, and flow rate for air samples
- ◆ Place of collection
- ◆ Analyses requested
- ◆ Comments

6.3 CLEANING SAMPLE COLLECTION EQUIPMENT AND CONTAINERS

Sample collection equipment and containers must be free of all analytes of interest and potential interferences. Where possible, disposable sample collection equipment and sample containers are used for collecting and transporting chemical agent-related waste samples. Equipment and containers intended for reuse are cleaned according to laboratory cleaning procedure WD-C METHOD: CL-021R "General Labware Washing and Maintenance." Between uses, scoops, shovels or other soil sample collection equipment are cleaned using a soap and water wash followed by a triple rinse with distilled water. Spent cleaning liquids are collected in drums designated and managed as potential chemical agent-related waste.

6.4 COLLECTING LIQUID SAMPLES

Liquid chemical agent-related wastes may include spent bleach and caustic decontamination solutions, IRP wastes, investigation derived waste (IDW), and other miscellaneous liquids. Liquid chemical agent-related wastes are typically stored in 55-gallon drums or large storage tanks. Where possible, liquid wastes have been segregated into waste streams based on the source of the waste, chemical agent exposure, and type of decontamination procedure used.

Generally, one sample is collected per drum or container of liquid waste. In the case of homogeneous liquid wastes being transferred from a large storage tank to multiple 55-gallon drums (a single "batch"), two samples (one at the beginning and another at the end of the transfer process) are considered sufficient. A rinse blank is collected if the sample collection equipment has been previously used.

Collecting liquid waste samples is described in WD-C METHOD CL-055R "Sampling Liquid Wastes." A Composite Liquid Waste Sampler (COLIWASA) is commonly used to collect free-flowing liquids and slurries from drums, shallow open tanks, pits, etc. Other acceptable liquid sample collection devices include the glass thief and the bailer. At the laboratory's discretion, individual phases that may exist within a sample may be separated by sample collection

personnel (noting the relative volumes of each phase in the original sample) or, preferably, a representative sample delivered to the laboratory for processing and analysis.

Liquid samples designated for chemical agent analysis are collected into clean glass containers. Samples are delivered to the laboratory as soon as possible as described in Section 6.11. Sample collection criteria is summarized in Table 6-1.

6.5 COLLECTING SOIL AND SOLID SAMPLES

Soil and solid chemical agent-related wastes may include soils related to spilled materials or any other soil from miscellaneous sources. Soils and solids generated during planned restoration activities are sampled and analyzed as part of the IRP.

The waste generator usually determines the number and location of samples to be collected with input from DEP and sample collection personnel. A rinse blank is collected if re-useable sample collection equipment is used. Collecting soil and solid waste samples is described in WD-C METHOD CL-057R "Sampling Soils and solids." Sample collection equipment must be free of analyte contamination and could include a stainless steel spoon, scoop, auger, and/or shovel.

Soil samples designated for chemical agent analyses are collected into clean glass containers. Samples are delivered to the laboratory as soon as possible as described in Section 6.11. Sample collection criteria are summarized in Table 6-1.

6.6 AIR MONITORING OF SOLID SAMPLES

Solid chemical agent-related wastes may include decontaminated solid test items, gloves and other project wastes, ventilation system wastes (including chemical agent contaminated pre-filters, high efficiency particulate air filters, plenums, duct work and activated carbon filters), IRP wastes, IDW, and other miscellaneous solid items (not including soils). Where possible, solid wastes are segregated into waste streams based on the source of the waste, chemical agent exposure, and type of decontamination procedure used. Depending on the project requirements (see the WAP, Section 3.2), solids may be sampled and analyzed using either air monitoring methods or solid sampling followed by laboratory analysis.

Sampling for air monitoring is performed using one of the following methods:

- ◆ WD-C METHOD CL-022R "Sampling Solid Wastes with DAAMS
- ◆ WD-C METHOD CL-044R "Chemical Agent Monitoring using Field MINICAMS®
- ◆ WD-C METHOD CL-045R "Lewisite Monitoring using Field MINICAMS®
- ◆ WD-C METHOD CL-056R "Sampling Solid Wastes with Bubblers

Generally, one sample is collected per container of solid waste (bag, gondola, etc.). Bagged items are sampled individually before transfer into a barrel or other larger container. Dry solid waste samples are placed in a sealed container and the contents are allowed to equilibrate for at least four hours at a temperature of 21°C (70°F) or higher. Small items may be placed, and heated if necessary, in a plastic bag having a minimum thickness of 2 MIL (~50 µm). Larger

items may be placed, and heated if necessary, in a roll-off or gondola and sealed with a tarp and packing tape. Following the equilibration period, the air surrounding the item in the container is sampled using a solvent-filled bubbler apparatus, Depot Area Air Monitoring System (DAAMS) tube sampler or MINICAMS[®] as described in the methods listed above. Sample analysis using MINICAMS[®] is performed at the location of the solid waste. Following sample collection, bubblers and DAAMS tubes are sealed, labeled with a unique sample number, and delivered to the laboratory for analysis. Sample collection criteria are summarized in Table 6-1.

Solid samples obtained for subsequent laboratory preparation and analysis must be collected in a representative manner in accordance with a DEP-approved sampling plan. The sampling plan must outline the sampling objectives, sample collection procedures, number and location of samples, required analyses for each sample, etc. A rinse blank is collected if re-useable sample collection equipment is used. Samples are generally collected into clean glass containers and delivered to the laboratory as soon as possible as described in Section 6.11.

6.7 COLLECTING FIELD QC SAMPLES

Field QC samples are intended to provide a measure of the cleanliness and representativeness of the sample collection activities. For chemical agent sample collection activities, field blanks, rinse blanks and/or duplicate samples may be required (see Table 6-2).

Field Blanks are used to detect possible contamination in the sample collection system. They are generally used when off-gas samples are collected using MINICAMS[®], DAAMS, or bubblers. Generally, one rinse blank is collected per sample collection lot (samples collected from the same waste description at the same time).

Rinse blanks are prepared by aspirating a known volume of background air using the same method as for samples. Rinse blanks are required when sample collection equipment (such as non-disposable Coliwass and bubblers) is cleaned and reused. Generally, one rinse blank is collected per sample collection lot (samples collected from the same waste description at the same time). Rinse blanks are prepared by running an analyte-free solution through sample collection equipment after cleaning but prior to sample collection. The rinse blank is analyzed and used to determine the effectiveness of equipment cleaning procedures.

Sample duplicates are required for liquids or soils when a new or unknown waste source is collected. Generally, one duplicate is collected per sample collection lot (samples collected from the same waste description at the same time). Sample collection personnel may also collect sample duplicates in order to accurately characterize complex matrices. A sample duplicate is simply a repeat of the sample that is sent to the laboratory to see whether the original sample results can be repeated.

Field spike samples (also known as QP Samples) are required when air sampling using bubblers or DAAMS. Generally, one field spike sample is collected per sample collection lot (samples collected from the same waste description at the same time). Field spike samples are prepared in the laboratory by adding a known amount of analyte to the bubbler or DAAMS tube. The spiked sample is taken to the field, aspirated with clean air at the monitoring location, and returned to the laboratory for analysis.

6.8 MAINTAINING INTERNAL CHAIN-OF-CUSTODY

To ensure integrity of compliance samples, sample collection personnel must be able to trace possession and handling of samples from the time of collection through delivery to the laboratory. A sample is considered to be under a person's custody if it is in the individual's physical possession, in the individual's sight, secured in a tamper-proof way by that individual, or secured in an area restricted to authorized personnel. A completed chain-of-custody (COC) record (see Exhibit 6-1) must accompany each sample or group of samples. External COC procedures are described in WD-C METHOD: ADM-018R "Maintaining Custody for Regulated Waste Samples."

6.9 REQUESTING LABORATORY ANALYSIS

Analyses to be performed on each sample must be clearly indicated on the COC or other documentation (see Exhibits 6-1 and 6-2). The analysis request documentation should include the following information:

- ◆ Type of analysis being requested
- ◆ Name, location, and phone number of sample requestor or contact
- ◆ Project and/or site description
- ◆ Sample identification (must be consistent with the sample containers)
- ◆ Sample matrix (liquid, soil, oil, etc.)
- ◆ Sample collection date and time
- ◆ Comments

6.10 NOTIFICATION OF SAFETY CONCERNS

Samples and accompanying paperwork must be adequately labeled to indicate any known or potential hazards such as flammability, corrosivity, toxicity, radioactivity, etc. Collection personnel and laboratory receiving personnel are responsible to communicate safety concerns to laboratory management and to laboratory personnel so that appropriate precautions can be taken during sample handling, storage, and disposal.

6.11 TRANSPORTING SAMPLES

Samples should be delivered to the laboratory as soon as possible after collection to ensure adequate time for analysis. Samples that cannot be delivered immediately to the laboratory must be held securely under documented control until delivery to the laboratory. Samples that cannot be delivered to the laboratory within 30 minutes should be stored and transported on ice to avoid degradation. A completed COC form must accompany samples and analysis request as described in Sections 6.8 and 6.9 above. The laboratory Sample Coordinator (or designated alternate) has the responsibility to reject samples at check-in for improper sample containers, incomplete paperwork, improper temperature at the time of receipt, or any other sample problem. The sample requestor will be notified immediately upon recognition of these problems.

Table 6-1. Summary of Chemical Agent-Related Waste Sample Collection Criteria.

Matrix	Waste Streams	Sample Collection Devices	Sample Container	Collection Frequency
Liquid Wastes	<ul style="list-style-type: none"> ▪ Spent decontamination solutions ▪ IRP liquid wastes ▪ IDW liquid wastes ▪ Miscellaneous liquid wastes 	<ul style="list-style-type: none"> ▪ Glass Coliwasa ▪ Glass thief ▪ Bailer ▪ Other as appropriate 	Glass	1 per drum 2 per batch
Soil Wastes	<ul style="list-style-type: none"> ▪ Spill materials ▪ Miscellaneous soil wastes 	<ul style="list-style-type: none"> ▪ Spoon ▪ Scoop ▪ Shovel ▪ Auger ▪ Other as appropriate 	Glass	Project specific
Solid Wastes (for air monitoring)	<ul style="list-style-type: none"> ▪ Decontaminated test items ▪ Project wastes ▪ Ventilation systems wastes ▪ IRP solid wastes ▪ IDW solid wastes ▪ Miscellaneous solid wastes 	<ul style="list-style-type: none"> ▪ MINICAMS[®] ▪ Bubbler apparatus ▪ DAAMS apparatus 	Bubbler or DAAMS tube	1 per container ¹
Solid Wastes (for extraction)	<ul style="list-style-type: none"> ▪ Miscellaneous solid wastes 	<ul style="list-style-type: none"> ▪ Spoon ▪ Scoop ▪ Shovel ▪ Auger ▪ Other as appropriate 	Glass	Project specific

¹ For MINICAMS[®], one sample consists of three cycles

Table 6-2. Summary of Field QC Sample Collection Requirements.

Matrix	Waste Streams	Blank Requirements	Field Duplicate Requirements	Other Field QC Requirements
Liquid Wastes	<ul style="list-style-type: none"> ▪ Spent decontamination solutions ▪ IRP liquid wastes ▪ IDW liquid wastes ▪ Miscellaneous liquid wastes 	One rinse blank per sample collection lot when sample collection equipment is reused	One field duplicate per sample collection lot when a new or unknown waste source is collected	None
Soil Wastes	<ul style="list-style-type: none"> ▪ Spill materials ▪ Miscellaneous soil wastes 	One rinse blank per sample collection lot when sample collection equipment is reused	One field duplicate per sample collection lot when a new or unknown waste source is collected	Consult with the laboratory to determine if extra sample is required for matrix spikes
Solid Waste (for air monitoring)	<ul style="list-style-type: none"> ▪ Decontaminated test items ▪ Project wastes ▪ Ventilation systems wastes ▪ IRP solid wastes ▪ IDW solid wastes ▪ Miscellaneous solid wastes 	One field blank per sample collection lot	Not applicable	For bubblers and DAAMS samples: one field spike sample (also known as QP sample) per sample collection lot
Solid Waste (for extraction)	<ul style="list-style-type: none"> ▪ Miscellaneous solid wastes 	One rinse blank per sample collection lot when sample collection equipment is reused	One field duplicate per sample collection lot when a new or unknown waste source is collected	Consult with the laboratory to determine if extra sample is required for matrix spikes


Exhibit 6-1. Example of COC/Analysis Request Form for Liquids and Soils.

CHAIN-OF-CUSTODY / ANALYSIS REQUEST										RCRA Hazardous Waste							
DPG CHEMICAL TEST DIVISION Combined Chemical Test Facility Dugway, Utah Phone: 522-5137					Requestor: _____ Office: _____ Phone: _____ XO: _____ CC: _____ Project: _____					Special Instructions: _____							
SAMPLE ID (1)	SAMPLE DATE	SAMPLE TIME	MATRIX (2)	# OF CONT.	AGENT ANALYSIS												COMMENTS
					G A	G B	G D	G F	H D	V D	X L						
1.																	
2.																	
3.																	
4.																	
5.																	
6.																	
7.																	
8.																	
(1) Sample ID includes barrel number, bag number, and/or other identification (2) Matrix = liquid, soil, bubbler, DAAMS, other (specify)																	
FIELD CHAIN-OF-CUSTODY								LABORATORY CHAIN-OF-CUSTODY									
Sampled By: _____				Received By: _____				Delivered to Lab By: _____						Received By: _____			
				Date/Time: _____										Date/Time: _____			
Relinquished By: _____				Received By: _____				Relinquished By: _____						Received By: _____			
				Date/Time: _____										Date/Time: _____			
Relinquished By: _____				Received By: _____				Relinquished By: _____						Received By: _____			
				Date/Time: _____										Date/Time: _____			
Relinquished By: _____				Received By: _____				Relinquished By: _____						Received By: _____			
				Date/Time: _____										Date/Time: _____			

Exhibit 6-2. Example of Request/Analysis Report Form for MINICAMS®.


MINICAMS REPORT FOR HAZARDOUS WASTE MONITORING			
Sample Information			
Generator Name: Location: Extension:		Return Report to: Location: Extension:	
Description of Contaminating Activity:		Date:	
Agent(s) Used (check all that apply) <input type="checkbox"/> GA <input type="checkbox"/> HD <input type="checkbox"/> GB <input type="checkbox"/> VX <input type="checkbox"/> GD <input type="checkbox"/> L <input type="checkbox"/> GF <input type="checkbox"/> Other:		Decontamination Procedure Used: <input type="checkbox"/> Hot water <input type="checkbox"/> Flame temp: <input type="checkbox"/> Steam <input type="checkbox"/> Oven: hours: <input type="checkbox"/> Solvent: type: <input type="checkbox"/> Other:	
Description of Waste Item(s):			
Unique Waste ID:			
Specific Instructions/Additional Information:			
Generator Signature:		Date:	
MINICAMS Summary (attach original printout to this form)			
Agent			
Name of Operator			
Date			
MINICAMS ID:			
Location			
Start/End Time	/	/	/
Start/End Flow (mL/min)	/	/	/
Start QC Results (1/2/3)	/ /	/ /	/ /
End QC Results (1/2/3)	/ /	/ /	/ /
QC Standard ID			
Amount Injected (µL)			
Blank Result (Average)			
Sample Result (Average)			
Above 1TWA (yes or no)			
Comments			
Operator (init/date)			
Supervisor (init/date)			
QC Reviewer (init/date)			

Exhibit 6-2. Example of Request/Analysis Report Form for DAAMS.



Data Analysis Sheet

Method: Solid Sorbent Tube



ANALYST: DATE ANALYZED: 12/8/00 INSTRUMENT: QC/4 barcode 40000 AGM/TEST ID: QA

SAMPLE NO./ID	ID#	TUBE ID#	DEEPWELL (L/min)			SAMPLING TIME			EXTRACTION TIME (min)	FLASK AGLA	ANALYST	VOLUME (mL)	TEMPERATURE (°C)	LOCATION IN LAB
			START	END	AVERAGE	START (min)	END (min)	AVERAGE (min)						
001121a	Q01	11111	0.4500	0.4500	0.4500	11.40	12.40	120	1.00	0.00	0.00	0.00	25.00	Location A
001121a	Q01	40000	0.4500	0.4500	0.4500	11.40	12.40	120	1.00	0.00	0.00	0.00	25.00	Location A
001121a	1	11004	0.4500	0.4500	0.4500	11.40	12.40	120	0.00	0.00	0.00	0.00	25.00	Location A
001121a	2	40000	0.4500	0.4500	0.4500	11.40	12.40	120	0.00	0.00	0.00	0.00	25.00	Location A
001121a	3	10000	0.4500	0.4500	0.4500	11.40	12.40	120	0.00	0.00	0.00	0.00	25.00	Location C
001121a	4													
001121a	5													
001121a	6													
001121a	7													
001121a	8													
001121a	9													
001121a	10													
001121a	11													
001121a	12													
001121a	13													
001121a	14													
001121a	15													
001121a	16													
001121a	17													
001121a	18													
001121a	19													
001121a	20													
001121a	Q02	11111	0.4500	0.4500	0.4500	11.40	12.40	120	1.00	0.00	0.00	0.00	25.00	Location A
001121a	Q02	11111	0.4500	0.4500	0.4500	11.40	12.40	120	1.00	0.00	0.00	0.00	25.00	Location A

RED Red Unchecked

Shirley Fleming General
Wine Team Time Control
National Institute of Standards and Technology

Data Entry Worksheet: 12/14/00

WDC 1700-4801
Revision 1
10/1/2000

7.0 GENERAL LABORATORY PROCEDURES

When samples are delivered to the laboratory, designated receipt personnel ensure that sample collection operations have been properly conducted and clearly documented. Receiving personnel must correctly document testing requirements and other information (such as required test methods, turnaround time, required sensitivity, and safety concerns) to analytical personnel.

This section describes the requirements for receiving and handling chemical agent-related waste samples. Sections 7.1 through 7.3 describe laboratory sample receipt, the storage and distribution of samples within the laboratory, and sample disposal, respectively. Section 7.4 discusses cleaning procedures for labware and sample collection equipment. Section 7.5 describes obtaining reagents, supplies, and services.

7.1 LABORATORY SAMPLE RECEIPT

Samples should be transported to the laboratory as soon as possible after sampling (see Section 6.0, Sample Collection). Chemical agent-related waste samples are received only by the laboratory Sample Coordinator (or designated alternate coordinators if the Sample Coordinator is unavailable). Sample receipt and handling procedures are detailed in WD-C METHOD ADM-013R "Laboratory Sample Receipt and Login."

Upon sample receipt, the Sample Coordinator will sign the COC (keeping the original), inspect the sample containers, review all accompanying documentation, prepare the necessary receiving records, and assign each sample a unique sequential laboratory sample identification number. The laboratory sample identification number is recorded in the sample receiving logbook (see Exhibit 7-1) along with any comments on the condition of the sample and the sample container. All samples, sub-samples, extracts, digestates, or other fractions derived from a sample will be labeled with the unique sample number assigned during sample receipt.

7.2 SAMPLE STORAGE AND DISTRIBUTION

Although no specific maximum holding time has been determined for chemical agent-related wastes, recommended holding times are listed in Table 7-1. Analysis of sample extracts usually occurs within 7 days of sample preparation. Bubbler samples are usually prepared and analyzed within 7 days of sample collection. Waste samples are stored at a temperature of ≤ 6.0 °C but above freezing while awaiting analysis. Samples removed from the refrigerator for analysis are returned as soon as possible.

The Sample Coordinator distributes samples to the analysts. Analytical personnel are alerted, in writing, of any special analytical or handling requirements as well as turnaround requirements.

7.3 INTERNAL CHAIN-OF-CUSTODY

Laboratory personnel must be able to trace possession and handling of samples inside the laboratory. A sample is considered to be under a person's custody if it is in the individual's physical possession, in the individual's sight, secured in a tamper-proof way by that individual, or secured in an area restricted to authorized personnel. Internal chain-of-custody (COC) forms

(see Exhibit 7-2) are used to track samples in and out of secured storage areas.

7.4 SAMPLE DISPOSAL

Unless other arrangements have been made with the Sample Coordinator, all samples will be disposed of after analysis and review of the Analytical Report. Sample disposal is performed in accordance with applicable safety and environmental regulation as described in the DPG Waste Management Plan.

7.5 LABWARE CLEANING AND MAINTENANCE

All glass and reusable plastic labware is thoroughly cleaned before use to avoid contamination. The cleanliness of reusable labware is evaluated using method blanks. Labware should be rinsed, decontaminated, and placed in a suitable soaking solution (such as a mild soap solution) immediately after emptying so that residues are not allowed to dry onto the glassware. All containers or wash tubs should be clearly marked to indicate their contents and, if applicable, the return location.

Labware cleaning is described in WD-C METHOD CL-021R: "General Labware Washing and Maintenance." After cleaning, borosilicate and other glass products should be inspected for chipping, cracking, or other abnormalities. Glass labware exhibiting signs of damage will be removed from service until repaired or discarded. Labware should also be inspected to determine if unusual cleaning might be required. Procedures for cleaning oil, grease, silicone, or other residues are found in the labware cleaning methods.

7.6 SUPPLIES & SERVICES

The laboratory relies on many outside sources for supplies and services which impact analytical quality. Laboratory equipment and supplies are purchased to meet or exceed the requirements of the analytical methods. Standard solutions, reagents and other chemicals must meet or exceed the quality and purity standards specified in the QAPP methods. The preparation of all reagent and standards solutions must be clearly documented and provide traceability to the materials and procedures used.

Table 7-1. Recommended Analytical Methods, Containers and Sample Holding Times

Determination	Method Reference¹	Container²	Preservative for Liquid Samples³	Recommended Maximum Holding Time
Chemical Agents - GC/MS	CL-002R ⁴	G	<6°C but above freezing	Prepare: 14 days Analyze: 7 days
Lewisite - GC (liquids and solids)	CL-025R	G	<6°C but above freezing	Prepare: 14 days Analyze: 7 days
Lewisite - GC (bubblers)	CL-025R	Bubbler	<6°C but above freezing	Prepare: 7 days ⁵ Analyze: 7 days
Chemical Agents - Technicon	CL-027R	G	<6°C but above freezing	Analyze: 14 days
Chemical Agents - MINICAMS [®]	CL-044R	NA	NA	Field Analysis
Lewisite - MINICAMS [®]	CL-045R	NA	NA	Field Analysis
Chemical Agents - DAAMS	CL-052R	DAAMS Tube	<6°C but above freezing	Prepare: 7 days ⁵ Analyze: 7 days

- 1 Equivalent methods may be used if approved by the UDSHW.
2 Container for solid samples is generally 4-6 ounce clear wide-mouth glass jar or plastic bag.
3 Preservation for solid samples is generally cooling to 4°C.
4 CL-002R replaces CL-001R and CL-004R.
5 This value is tentative until an official holding time study can be completed.

°C Degrees Celsius
EPA U.S. Environmental Protection Agency
G Glass
NA Not Applicable
TCLP Toxicity Characteristic Leaching Procedure

Exhibit 7-1. Example of Sample Receiving Logbook Page.

SAMPLE RECEIVING LOG															
Lab Number	Receiving Information				Sample Information				Requestor Information			Laboratory Information			
	Date Recd.	Time Recd.	Deliv. By	Recd. By	# of Samples	Batch	Project	Sample ID	Sample Type	Name	Office	Phone	Analyses Desired	Sample Coord.	Date Complete
97-125															
97-126															
97-127															
97-128															
97-129															
97-130															
97-131															
97-132															
97-133															
97-134															
97-135															
97-136															
97-137															
97-138															
97-139															
97-140															
97-141															
97-142															
97-143															
97-144															
97-145															
97-146															
97-147															
97-148															
97-149															
97-150															

8.0 CALIBRATION PROCEDURES AND FREQUENCIES

Calibration is accomplished through the use of reference materials supplied by the Chemical Agent Standard Analytical Reference Materiel (CASARM) Program of the U.S. Army Chemical and Biological Defense Command (CBDCOM). The reference materials are stringently analyzed and certified by the CASARM Program. The program includes ongoing validation to ensure that reference material degradation does not occur. These reference materials are used throughout the military chemical-defense complex. Solutions derived from these reference materials are prepared at DPG and used to calibrate instrumentation. This system ensures that all measurements within the military complex are comparable and traceable to an accepted standard.

The sections below detail the calibration procedures used in the DPG chemical agent-related Hazardous Waste Analysis Program. Additional calibration information is found in the individual analytical methods listed in Section 2.0. Section 8.1 describes the handling of reference materials. Section 8.2 describes the calibration requirements for laboratory instrumentation, and Section 8.3 describes the calibration requirements for MINICAMS®.

8.1 HANDLING REFERENCE MATERIALS

Standards are prepared from CASARM in accordance with the requirements of WDTC Operating Procedure DP-0000-M-73. Generally, two analysts independently prepare two stock solutions. One solution is used to prepare working standards and the other is used to prepare verification standards. All manipulations and dilutions are recorded. All solutions must be traceable to the CASARM. All uses of solutions are recorded to ensure traceability.

8.2 CALIBRATING LABORATORY INSTRUMENTS

This section describes the calibration of laboratory instrumentation including stationary gas chromatographs and the Technicon AutoAnalyzer. Detailed calibration instructions are given in individual analytical methods.

Initial calibration is required for laboratory instrumentation within a method-specified time period if significant changes are made to the instrument, or if the calibration verification fails. In general, initial calibration is performed for each analyte with a minimum of four concentrations and an instrument blank. The linear or second order regression analysis of the calibration curve must result in an r^2 value (where r is the correlation coefficient) of at least 0.990. The calibration curve is verified with an initial calibration verification solution that must be recovered within $\pm 15\%$ of true value, unless specified otherwise in the analytical method.

Continuing calibration is performed by analyzing calibration check (CC) standards within each analytical run to ensure that the initial calibration is still valid. At a minimum, a CC is analyzed after every ten or fewer waste samples and/or after any standby period or other period of disuse. CC standards must be recovered within $\pm 20\%$ of true value, unless specified otherwise in the analytical method. If the CC fails, it is repeated. If it fails a second time, then an initial calibration must be performed.

8.3 CALIBRATING FIELD MINICAMS®

The calibration procedures for field MINICAMS® are detailed in WD-C METHOD CL-044R “Chemical Agent Monitoring (GA, GB, GD, GF, HD, and VX) using Field MINICAMS® and WD-C METHOD CL-045R “Chemical Agent Monitoring (Lewisite) using Field MINICAMS®. Initial calibration is required each time the MINICAMS® is moved to a new location, if significant changes are made to the instrument, or if the CC (“QC” sample) fails. Generally, the field MINICAMS® is calibrated for the analyte(s) of interest by first placing it in the calibration mode. A known amount (at or near the regulatory level) of standard is injected into the instrument during two successive cycles. The MINICAMS® will automatically calculate the average response factor from the two injections and store the new calibration. Initial calibration is verified by injecting a known standard prepared at two times the regulatory level. A result of between 1.5 and 2.5 times the known value ($\pm 25\%$) is considered satisfactory.

Continuing calibration is required after initial calibration, at the beginning and end of each run, and after every 10 hazardous waste samples. To perform a continuing calibration, a “QC” standard (prepared at or near the regulatory level) is injected into the instrument during the sampling period of the MINICAMS® cycle. A result of between 0.75 and 1.25 times the known value ($\pm 25\%$) is considered satisfactory. If the first “QC” fails, a second is injected. If the second “QC” also fails, corrective action should be taken as described in the method. If, following corrective action, a third “QC” fails, the MINICAMS® should be removed from service for repair or refurbishment.

9.0 LABORATORY QUALITY CONTROL

Method-specific laboratory QC measures are used to assure that the analytical process is in control. QC parameters may include rinse and method blanks (used to evaluate cleanliness), method blank spike (MBS) samples (used to evaluate accuracy), and method blank spike duplicate (MBSD) samples (used to evaluate precision). Data Quality Objectives (DQOs) for cleanliness, accuracy, and precision (Sections 9.1 through 9.3) are established to ensure that the data will support the objectives of the DPG waste analysis and management programs. Section 9.4 outlines the determination of analytical method performance. Sections 9.5 and 9.6 discuss determination of the method detection limit (MDL) and reporting limit (RL).

9.1 OBJECTIVES FOR CLEANLINESS

Cleanliness is defined as the absence of contamination in the field and laboratory. Field contamination is evaluated using field blanks and rinse blanks (Section 6.7). In general, field blanks are collected when off-gas samples are collected using MINICAMS[®], DAAMS, or bubblers. Rinse blanks are required for liquid and soil matrices when sampling equipment is being re-used. The concentration of all target analytes in the rinse blank should be less than the RL. Specific requirements for the sample collection are found in Section 6.0 and the individual sampling methods.

Laboratory contamination is evaluated using the method blank. In general, the concentration of all target analytes in the method blank should be less than the RL. Specific requirements for the preparation and evaluation of method blanks are found in the individual analytical methods. Results that do not meet the DQO for cleanliness require corrective action as described in Section 12.0. Cleanliness DQOs for chemical agent-related waste analysis are provided in Table 9-1.

9.2 OBJECTIVES FOR ACCURACY

Accuracy is a measure of the ability of the analytical method to achieve a known analytical result. For chemical agent-related wastes, accuracy is usually evaluated by analyzing a clean matrix sample (method blank) that has been spiked with known amounts of the target compounds. In some cases, matrix spike samples may also be indicative of method accuracy. Details on the preparation of MBS samples are found in the individual analytical methods.

Percent recovery (%R) for each MBS compound is calculated as:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where: SSR = spiked sample result
 SR = unspiked sample result (usually zero)
 SA = spike amount added to the sample

The %R for each compound, method, and matrix is compared with previous data using statistical QC charts. The result must be within the 99% confidence limits. In the absence of adequate statistical data for %R, an acceptance range of 80 - 120% will be used as a guide. Results that do not meet the DQO for accuracy require corrective action as described in Section 12.0. Accuracy DQOs for chemical agent-related waste analysis are provided in Table 9-1.

9.3 OBJECTIVES FOR PRECISION

Precision is a measure of the variability of the analytical method. For chemical agent-related wastes, precision is most often evaluated by comparing the results of the MBS and MBSD recoveries using the range (R) or the relative percent difference (RPD). In some cases, matrix spike duplicates may also be used to evaluate precision. R and RPD are calculated as:

$$R = |MBSR - MBSDR|$$
$$RPD = \left| \frac{2(MBSR - MBSDR)}{MBSR + MBSDR} \right| \times 100$$

where: MBSR = MBS percent recovery
MBSDR = MBSD percent recovery

Either R or RPD for each compound, method, and matrix is compared with previous data using statistical range QC charts. The result must be within the 99% confidence limits. In the absence of adequate statistical data for RPD, an acceptance limit of 20% will be used as a guide. Results that do not meet the DQO for precision require corrective action as described in Section 12.0. Precision DQO's for chemical agent-related waste analysis are provided in Table 9-1.

9.4 ANALYTICAL METHOD PERFORMANCE

Analytical method performance is defined in terms of accuracy and precision. Method accuracy and precision are determined during method development by preparing and analyzing at least eight mid-level (approximately 10-20 times the estimated MDL) replicate samples. Method performance is often determined in conjunction with the DPG Safety Air Monitoring precision and accuracy study.

Accuracy (percent recovery, %R) and Precision (relative standard deviation, RSD) are calculated using the following formulas:

◆ Method Accuracy

$$\%R = \frac{\text{average}}{\text{expected}} \times 100$$

where: average = average result
expected = true value

◆ Method Precision

$$RSD = \frac{S}{\text{average}} \times 100$$

where: s = standard deviation of replicate results
average = average result

Results of the method performance studies provide a basis for ongoing QC requirements as described above.

9.5 METHOD DETECTION LIMITS

The MDL is an estimate of the lowest level of an analyte that can be distinguished from noise. For chemical agent-related analyses, the MDL is experimentally determined at least annually by preparing and analyzing seven or more low level (1-5 times the estimated MDL) interference-free replicate samples. MDLs are often determined in conjunction with the DPG Safety Air Monitoring precision and accuracy study. For GC/MS analyses using the selected ion monitoring (SIM) mode, the MDL is determined using the least sensitive ion required for positive identification.

The MDL is calculated using the following formula:

$$MDL = t \times s$$

where: t = student's t statistic (2.896 for eight replicates)
s = standard deviation of replicate results

9.6 REPORTING LIMITS AND ACTION LEVELS

Unlike the interference-free standards prepared for determination of the MDL, field samples often contribute significant "noise" to the analytical procedure and the instrument response. The RL is defined as the lowest reportable analyte concentration for a particular sample given the MDL, matrix, extraction and dilution effects, interferences, analytical "noise," and other relevant factors. The RL is usually a factor of 2 to 20 times the MDL. Given the hazardous nature of chemical agents, RLs should be conservatively chosen to eliminate the chance for false negative results (a non-detect at the RL when analyte is actually present above that level). Analyte levels between the MDL and RL are reported with a "J" qualifier, estimated value. Hazardous wastes are not transported to the CHWSF if analytical results indicate that chemical agents are present above the action levels listed in Table 9-2.

Table 9-1. DQO's for Chemical Agent-Related Waste Analyses.

Analytical Method	Matrix	Cleanliness		Accuracy		Precision	
		Parameter	Criteria	Parameter	Criteria	Parameter	Criteria
CL-002R*	Liquids and Soils	MB	all target compounds <RL	MBS	%R within 99% CL	MBS/MBSD	R or RPD within 99% CL
CL-056R/025R	Liquids, Soils, and Bubblers	MB	all target compounds <RL	MBS	%R within 99% CL	MBS/MBSD	R or RPD within 99% CL
CL-027R	Bubblers	MB	all target compounds <RL	MBS	%R within 99% CL	MBS/MBSD	R or RPD within 99% CL
CL-044R	Solids	MB	all target compounds < 0.5 HL	"QC" Sample	%R within $\pm 25\%$	NA	NA
CL-045R	Solids	MB	all target compounds < 0.5 HL	"QC" Sample	%R within $\pm 25\%$	NA	NA
CL-022R/052R	DAAMS	MB	all target compounds < 0.5 HL	"QP" Sample	%R within $\pm 25\%$	QL/QL	RPD <20%

*Method CL-002R replaces CL-001R and CL-004R.

%R	Percent Recovery
CL	Confidence Limit
DAAMS	Depot Area Air Monitoring System
HL	Hazard Level (TWA for air)
MB	Method Blank
NA	Not Applicable
QC	Quality Control
QL	Quality Laboratory Sample
QP	Quality Plant Sample
R	Range
RL	Reporting Limit
RPD	Relative Percent Difference
TWA	Time Weighted Average

Table 9-2. Action Levels for Waste Characterization.

Matrix	Analytical Methods	Analyte	Action Level	Units
Liquid	CL-002R ⁽¹⁾ (GC, GC/MS) CL-025R (GC)	GA	0.02	mg/L
		GB	0.02	mg/L
		GD	0.02	mg/L
		GF	0.02	mg/L
		HD	0.2	mg/L
		HN-3	0.2	mg/L
		VX	0.02	mg/L
		Lewisite	0.2	mg/L
Soils/Solids	CL-002R ⁽¹⁾ (GC, GC/MS) CL-025R (GC)	GA	MDL ⁽²⁾	mg/kg
		GB	MDL ⁽²⁾	mg/kg
		GD	MDL ⁽²⁾	mg/kg
		GF	MDL ⁽²⁾	mg/kg
		HD	MDL ⁽²⁾	mg/kg
		HN-3	MDL ⁽²⁾	mg/kg
		VX	MDL ⁽²⁾	mg/kg
		Lewisite	MDL ⁽²⁾	mg/kg
Air	CL-044R (MINICAMS) CL-045R (MINICAMS) CL-056R/CL-025R (Bubblers) CL-022R/CL-052R (DAAMS)	GA	0.00002 ⁽³⁾	mg/m ³
		GB	0.00002 ⁽³⁾	mg/m ³
		GD	0.000006 ⁽³⁾	mg/m ³
		GF	0.00002 ⁽³⁾	mg/m ³
		HD	0.0006 ⁽³⁾	mg/m ³
		HN-3	0.0006 ⁽³⁾	mg/m ³
		VX	0.000002 ⁽³⁾	mg/m ³
		Lewisite	0.0012 ⁽³⁾	mg/m ³

⁽¹⁾ Method CL-002R replaces CL-001R and CL-004R.

⁽²⁾ Risk-based action levels have not been determined for soils and solids. The MDL is specific to an analytical instrument (such as GC/MS-SIM, GC/FID, and GC/FPD). The MDL will be used for the action level until action levels are promulgated by UDSHW. The CHWSF may accept F999 and P999 wastes only if associated chemical agent MDL studies are up to date (see Section 9.5).

⁽³⁾ Air action levels are 2-5 times lower than the recognized risk-based safety air monitoring levels. These levels apply for Igloo G monitoring or when air monitoring is the primary analysis method for hazardous waste acceptance to the CHWSF (such as for solid test-related debris, ventilation filters, etc.).

mg/kg milligrams per kilogram
mg/L milligrams per liter
mg/m³ milligrams per cubic meter

10.0 ANALYTICAL DATA MANAGEMENT

The purpose of the QA program described in this QAPP is to ensure that only valid, reliable data are reported. In order to be reported, analytical data must meet the applicable QC requirements (see Section 9.0), and then be correctly recorded, reduced, reviewed, and reported. In addition, a subset of all reported data is subject to independent validation as described below. The process of generating valid and defensible analytical data includes the following:

- ◆ Data Recording (Section 10.1)
- ◆ Data Reduction (Section 10.2)
- ◆ Data Reporting and Review (Section 10.3)
- ◆ Data Validation (Section 10.4)

10.1 DATA RECORDING

The laboratory record system must produce unequivocal, accurate records that document all laboratory activities. The laboratory retains on record all original observations, calculations, derived data, calibration records, and a copy of the test report for at least five years. The laboratory also maintains all hardware and software necessary for the historical reconstruction of data for five years. The record-keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes.

The history of the sample within the laboratory must be readily understood through the laboratory documentation. The records for each test must contain sufficient information to allow the historical reconstruction of all laboratory activities related to analytical data produced. Where possible, checklists and/or forms (electronic or printed) are used to ensure that data are recorded and presented accurately and consistently. Records must be legible and give sufficient detail to enable an independent reviewer to:

- ◆ Reconstruct the sequence of events
- ◆ Reconstruct calculations
- ◆ Establish that key steps were completed
- ◆ Establish that method specified recording requirements were met
- ◆ Establish that the record is complete

Data generated within the laboratory must be documented according to scientifically acceptable standards. These include:

- ◆ Data must be recorded at the time it is generated
- ◆ Data must be recorded by the generator of the data, or a direct observer
- ◆ Errors are crossed out with a single straight line
- ◆ Corrected data is entered, initialed, and dated
- ◆ No erasures or correction fluid is allowed
- ◆ Hand-entered data are recorded with permanent ink pen
- ◆ Data which are recorded or generated electronically shall be printed out, signed, and dated by the operator (on the cover page if the report is stapled or bound)

- ◆ Each page of a multi-page record or report must be numbered to show the page number and the first page must state the total number of pages in the record or report
- ◆ Fields in forms that are not used are lined through with a single diagonal line or noted as Not Applicable (NA).
- ◆ If electronic data are to be included in a logbook, the printout is secured (taped, stapled, or pasted) in the logbook, then signed. The signature and date must cross both the print-out and the page to which it is secured

Records that are stored on computers will have hard copy or write-protected backup copies. Archived records are protected against fire, theft, loss, environmental deterioration, and in the case of electronic records, electronic or magnetic sources.

10.2 DATA REDUCTION

Data reduction is the process of converting an analytical signal or response to a reportable result. Depending upon the test and instrumentation involved, data are reduced and reported using both manual and automated procedures. If the data are manually processed and reported by an analyst, all steps in the computation are recorded for review including equations used and the source of input parameters such as response factors, dilution factors, and calibration data. The analyst signs and dates each page of calculations and data in a bound logbook for review and verification purposes.

Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data, the laboratory will ensure that computer software is documented and adequate for use. Before releasing the data to the reporting system, the analyst must verify that information such as the sample numbers, calibration information, dilution information, and detection limits have been correctly entered. A hard copy printout of all computer-generated data is obtained for data review and verification purposes.

Conversion of analyte signal to analyte concentration is usually performed by comparison to a calibration standard or calibration curve. Since the sample may have gone through digestion, extraction, dilution, and/or concentration prior to analysis, the reporting concentration may be determined using the following formula:

$$\text{Reporting Concentration} = \frac{Q \times df \times Vf}{Vi} \times U$$

where: Q = concentration determined by comparison the calibration curve (typically $\mu\text{g/L}$, mg/L , or mg/Kg)
df = ultimate dilution factor (if needed)
Vf = final extract volume (mL)
Vi = initial sample volume (mL) or weight (g)
U = unit conversion factor, such as μg to mg (if needed)

10.3 DATA REPORTING AND REVIEW

Final analytical results are clearly annotated on the analytical data or data summary. An analysis report is prepared using the final results from associated analytical data. The following information should be included on the analysis report:

- ◆ Requestor information
- ◆ Sample information
- ◆ Analytical results
- ◆ Method used
- ◆ Date analyzed
- ◆ Data qualifiers, if any (such as “J” for estimated value)
- ◆ Approval signatures

Key analytical information is assembled in an analytical file for easy retrieval and review. Data packages should include the following, where applicable:

- ◆ A copy of the Analysis Report
- ◆ Chain-of-Custody/Analysis Request form
- ◆ Relevant sample collection information
- ◆ Other sample-related information
- ◆ Analytical summaries
- ◆ A QC summary
- ◆ A copy of run log or sequence summary
- ◆ The calibration curve raw data (including chromatograms)
- ◆ A graph of the calibration curve
- ◆ The raw data for samples and QC samples (including chromatograms)
- ◆ A photocopy or reference to standard preparation logbook page(s)
- ◆ A photocopy or reference to reagent preparation logbook page(s)
- ◆ A photocopy or reference to other applicable laboratory records
- ◆ Other associated analytical information

100% of the assembled analytical files are submitted to another qualified individual within the laboratory for peer review as described in WD-C METHOD: ADM-022R “Peer Review of Analytical Data.” During this review, a peer reviewer checks the data packages for completeness and ensures that the resulting data comply with method requirements. Upon approval, the peer reviewer signs and dates the Analytical Data Checklist and submits the analytical record to the QC Chemist for final review and approval as described in WD-C METHOD: ADM-019R “Quality Control Review of Analytical Data.”

Issued Analysis Reports will remain unchanged. Amendments to an Analysis Report after issuance are made only in the form of a further document that clearly states *AMENDED REPORT*. A cover letter should indicate the date and purpose of the amendment and be signed by all original signatories to the original report.

10.4 DATA VALIDATION

At least 10% of the chemical agent-related waste analyses are independently validated as described in WD-C METHOD: ADM-012 "Validation of Analytical Data." This is accomplished by validating every tenth RCRA sample. Validation is performed under the direction of DEP personnel. During the validation process, analytical records are checked for completeness as well as compliance with this QAPP and applicable methods. Validation personnel will ensure that all computer calculations and manipulations are appropriate and correct.

11.0 LABORATORY QUALITY ASSESSMENT

Quality assessment is the process of using internal and external measures to determine the quality of the data produced by the laboratory. Laboratory quality assessment is accomplished using control charts and proficiency test samples, as well as internal and external audits and reviews. Sections 11.1 and 11.2 below describe the use of control charts and proficiency test samples to assess laboratory performance. Sections 11.3 and 11.4 describe internal and external audits. Section 11.5 describes the management's annual system review.

11.1 CONTROL CHARTS

Control charts with control limits are statistical tools for monitoring the performance of laboratory QC parameters such as CC standards, MBS samples, and MBSD samples. Generally, control limits are used internally to evaluate and improve system quality. Where available, method-specified QC acceptance limits are used to determine data acceptability for reporting purposes.

Two types of control charts may be used in the laboratory: an accuracy means chart for CC and MBS percent recovery (Figure 11-1), and a precision range chart for MBS/MBSD relative percent difference (Figure 11-2). The control limits are set at ± 3 standard deviations from the mean for accuracy and precision. Control limits are updated every 20-30 data points.

11.2 PROFICIENCY TEST SAMPLES

The CBDCOM's CASARM QA Team provides a Proficiency Testing Program. DPG participates in all available rounds of this program. The QC Chemist reviews proficiency test reports. Corrective actions are undertaken for any missed analytes (see Section 12.0).

11.3 INTERNAL AUDITS

The QC Chemist performs, or arranges for, audits to verify that waste-related analytical activities continue to comply with the requirements of the quality system. Persons who are trained and qualified as auditors carry out these audits on a semi-annual basis. Auditors must be organizationally independent of the activity to be audited.

During the internal audits, sample collection, handling, analysis, and reporting activities are

evaluated according to the requirements of the quality system and methods. Internal quality system audits should include the following areas:

- ◆ Sample collection procedures and documentation
- ◆ COC procedures and documentation, including sample identification
- ◆ Laboratory sample receiving procedures and documentation
- ◆ Analytical procedures and documentation, including sample preparation, instrument calibration, and data reduction
- ◆ QC procedures and documentation
- ◆ Data review procedures
- ◆ Method validation for any new procedures
- ◆ Sample storage
- ◆ Data packaging or reporting procedures

The goal of the audit is to detect any deviations from acceptable practices and procedures so that corrective action can be taken. When an audit finding cast doubt upon the correctness or validity of any test results, the laboratory will take immediate corrective action and immediately notify any client whose work may have been affected. Audit-related findings will be addressed through the corrective action system (Section 12.0).

11.4 EXTERNAL AUDITS

From time to time, data users (such as DEP and other sample requesters) and regulators (such as the State of Utah DSHW) will desire to audit the chemical agent-related laboratory activities at CCTF. The laboratory will cooperate, to the fullest extent possible, in assisting with these audits.

All audit-related activities will be coordinated through the Analytical Branch Chief. While in the laboratory, auditors will be accompanied by CCTF staff to maintain confidentiality and security. Audit-related findings will be addressed through the corrective action system (Section 12.0).

11.5 MANAGEMENT REVIEW

The Analytical Branch Chief will lead and coordinate an annual management review and evaluation of this QAPP to verify its suitability and effectiveness. The review team will include the Analytical Branch Chief and the Branch QC Chemist, as well as management representatives from CTD and DEP. Results of the review will be documented. Changes implemented based upon the review will be documented and verified.

The management review will include, but not be limited to, the following:

- ◆ Review and evaluation of the records of internal and external audits of the laboratory quality system
- ◆ Evaluation of external influences such as additional work, new technology, changing or new regulations, organizational changes, etc.
- ◆ Evaluation of the adequacy of personnel, facilities, and equipment

◆ Review of recommended courses of action

The QC Chemist is responsible for evaluating and responding to the recommendations generated by the management review. Audit-related findings will be addressed through the corrective action system (Section 12.0).

Figure 11-1. Mean Control Chart for Accuracy.

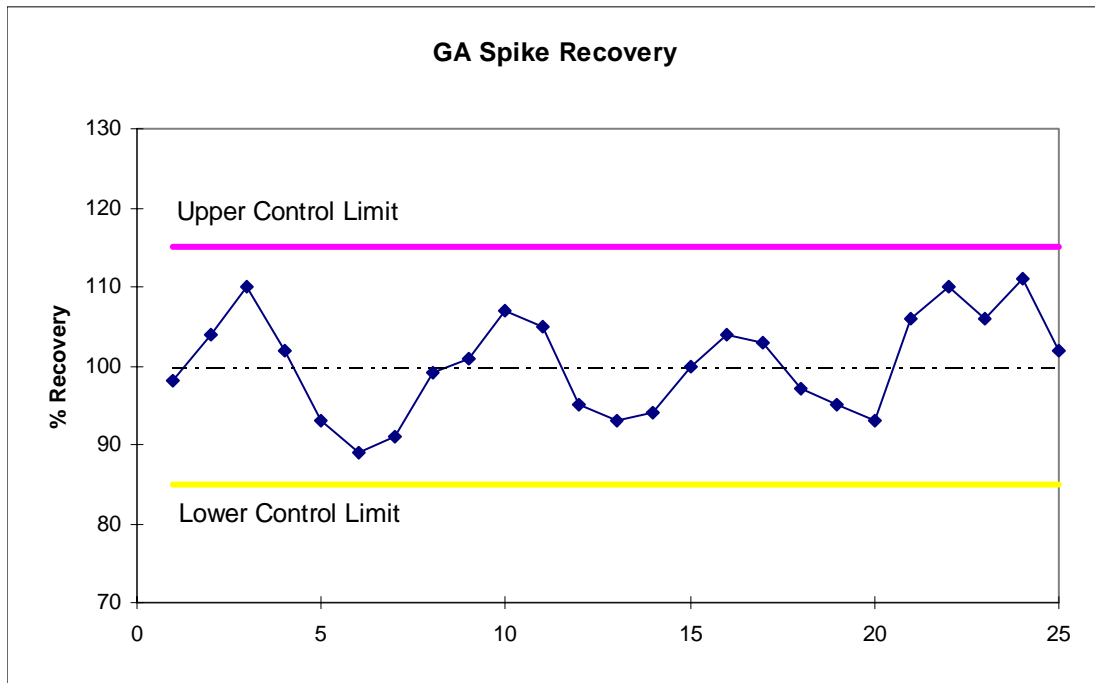
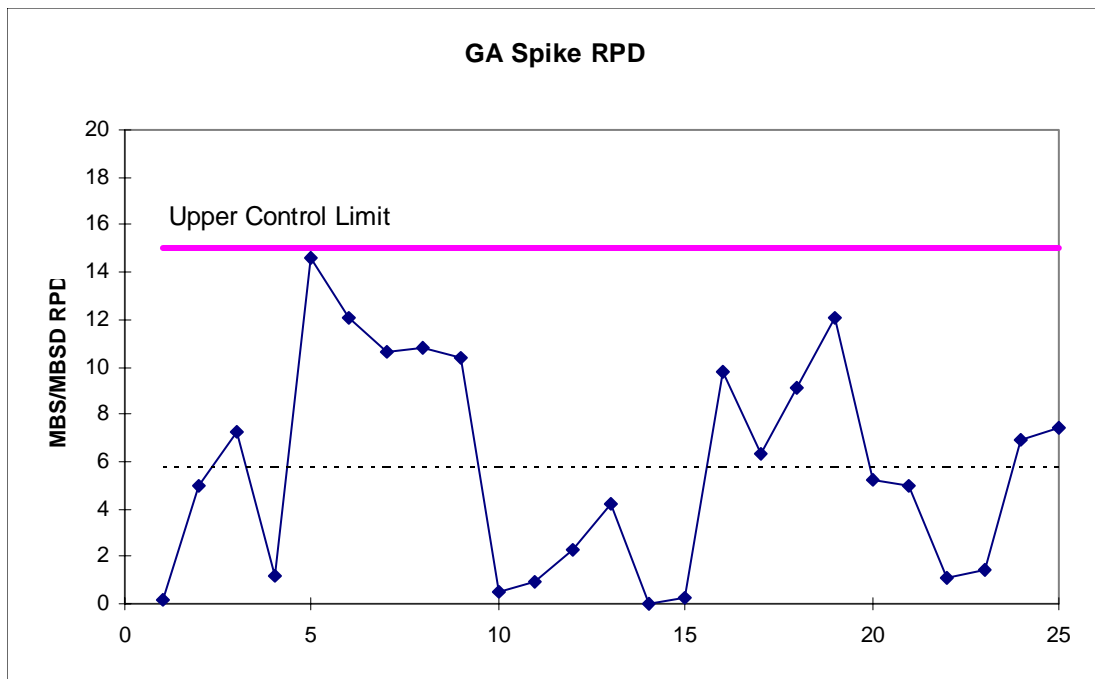


Figure 11-2. Range Control Chart for Precision.



12.0 CORRECTIVE ACTION

The laboratory has a formal system for initiating and implementing corrective action. Corrective action and follow-up are powerful tools for continuous improvement within the laboratory. Specific corrective action procedures depend on the nature of the discrepancy or out-of-control situation. Ultimately, the QC Chemist is responsible for identifying and correcting systemic quality problems within the laboratory. Individuals working in the laboratory, however, must be familiar with all QC policies and procedures and bring discrepancies to the attention of the QC Chemist or management personnel.

For guidance purposes, two types of analytical problems have been identified below; bench analytical problems (Section 12.1) and administrative or systemic problems (Section 12.2). The chemist or supervisor often will solve bench analytical problems immediately without initiating a formal corrective action report (CAR). Administrative or systematic corrective action usually requires the use of a formal CAR.

12.1 BENCH ANALYTICAL PROBLEMS

Bench analytical problems are those that may occur during sample analysis. These types of errors include failed calibration, failed continuing calibration, failed method spike recovery, etc. Many of these problems can and should be corrected at the time of analysis and do not require external documentation using the CAR.

All laboratory personnel should be aware of the specific QC requirements associated with their analytical responsibilities. Under no circumstances should data be released from the bench unless: (1) All QC results are within acceptable limits, or (2) The suspect data have been clearly qualified as to the nature of the discrepancy, the corrective actions which have been taken and the results of the corrective actions.

Corrective action is a function of the type or error encountered. Experienced analysts and supervisors should be consulted when trouble-shooting these types of problems. Possible corrective actions for bench analytical problems may include:

- ◆ Re-run failed QC sample and/or calibration standards
- ◆ Re-prepare and re-run QC sample and/or calibration standards and field samples
- ◆ Re-prepare and re-run field sample(s) (if feasible) associated with the failed calibration
- ◆ Perform routine instrument maintenance

12.2 ADMINISTRATIVE OR SYSTEMIC PROBLEMS

Administrative or systemic problems may include errors in sample receipt, holding time, sample preservation, data transcription, data reporting, performance evaluation results, etc. These types of errors are usually discovered during data review, internal audits, or external performance evaluation audits. They may also be brought to the attention of the laboratory by clients (i.e., customer complaints) or external auditors.

Administrative and systemic problems may be very significant and corrective actions must identify the root cause of the problem (insufficient resources, lack of training, no internal checks, etc.) and recommend possible solutions (improve resources, provide training, increase internal checks, etc.). This process is documented using a Corrective Action Form (see Exhibit 12-1). Every effort will be made to identify and resolve quality problems in an equitable and timely manner. As part of the corrective action process, the QC Chemist and laboratory management will review and recommend changes to the QAPP and methods, if necessary, to avoid similar problems in the future. When completed, CARs are signed and maintained by the QC Chemist.

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13.0 REFERENCES

Interim Guidelines and Specifications for Preparing Quality Assurance Program Plans (EPA, 1983)

Preparation Aids for the Development of Category 1 Quality Assurance Program Plans (EPA, 1991)

Interim Draft Requirements for Preparing Quality Assurance Project Plans (EPA, 1984)

Test Methods for Evaluating Solid Waste, Update III, SW-846 (EPA, 1997)

Preparation of Standard Solutions, WDTC Operating Procedure DP-0000-M-73

14.0 ACRONYMS AND DEFINITIONS

%R - Percent Recovery

°C - degree(s) Celsius

°F - degree(s) Fahrenheit

CO - Consent Order

COC - Chain-of-Custody

COLIWASA - Composite Liquid Waste Sampler

CPO - Civilian Personnel Office

CRD - Compliance and Restoration Division

CTD - Chemical Test Division

Calibration Check Standard - Analytical standard run in a specified sequence or time interval to verify that the calibration of the analytical system remains in control.

CAR - Corrective Action Report

CASARM - Chemical Agent Standard Analytical Reference Materiel

CBDCOM - Chemical and Biological Defense Command

CC - Calibration Check

CCL - Chemical Control Limit

CCTF - Combined Chemical Test Facility

Chemical Agent - Any of several highly toxic chemical compounds (including GA, GB, GD, GF, HD, L, and VX) that are intended for use in military operations

CL - Confidence Level

Cleanliness - The absence of contamination in the laboratory as measured by blanks.

Comparability - The degree an analysis performed by one laboratory agrees with an analysis performed on a similar sample by another laboratory.

Completeness - The degree to which an analysis or batch of analyses has met all other Data Quality Objectives.

Controlled Document - A document that is issued to personnel with a document tracking number.

DAAMS - Depot Area Air Monitoring System

Data Quality Objectives - Standards which the laboratory strives to maintain. They establish a goal or benchmark for laboratory performance.

Data Validation - An independent evaluation of an analyses' adherence to the analytical methods and quality assurance procedures.

Data Package - A set of records describing the complete history of a defined set of events (records) pertaining to a single laboratory sample lot.

Decontamination - The process of decreasing the amount of chemical agent on any person, object, or area by absorbing, neutralizing, destroying, ventilating, or removing chemical agents.

DEP - Directorate of Environmental Programs

Depot Area Air Monitoring System - Various solid sorbent tubes used on DPG to collect Safety Air Monitoring Samples from the headspace surrounding solids

Dilution Factor - The volume-to-volume ratio of a sample extract to a dilution of that extract which is analyzed

DPG - U.S. Army Dugway Proving Ground

DQO - Data Quality Objectives

DSHW - Division of Solid and Hazardous Waste

EPA - U. S. Environmental Protection Agency

Field Duplicate - Duplicate samples collected in the field to establish the overall precision of the sampling and analytical process. Duplicates are handled like routine samples in the laboratory.

Field QC Samples - Samples that provide a measure of the quality of the sampling activities.

Field Sample Lot - Twenty or fewer samples collected from the same waste description at one time (shift) by a single team of Sampling Personnel. Each field sample lot for liquid is accompanied by field QC samples including an FD and an equipment Rinse Blank when using non-disposable equipment.

Field Spike Sample - See QP Sample

GA - Tabun: Ethyl N,N-dimethylphosphoroamidocyanide, CAS 77-81-6, a nerve agent

GB - Sarin: Isopropyl Methylphosphonofluoridate, CAS 107-44-8, a nerve agent

GC - Gas Chromatography

GD - Soman: Pinacolyl Methylphosphonofluoridate, CAS 96-64-0, a nerve agent

GF - Cyclohexyl Methylphosphonofluoridate, CAS 329-99-7, a nerve agent

Hazard Level Standard - A calibration standard prepared at a concentration equivalent to the 8-hour time weighted average (TWA) exposure limit (assuming a given injection volume, flow rate, and cycle time). Also known as "initial calibration."

HD - Mustard, Distilled: Bis-2-chloroethyl sulfide, CAS 505-60-2, a blister agent

HL - Hazard Level

HN-3 - tris-2-chloroethylamine – Nitrogen Mustard

IDW - Investigation Derived Waste

Initial Calibration Verification Standard - A standard material, prepared independently from the calibration standards, which is used to verify a new set of calibration standards

IRP - Installation Restoration Program

Issuing - Distributing and controlling master copies of controlled documents

Laboratory Sample Lot - A laboratory sample lot consists of 20 or fewer samples. It is the maximum number of samples, up to 20, that can be manually processed through the method during a single time period, not to exceed 24 hours.

Lewisite - Dichloro-(2-chlorovinyl) arsine

Lot Number - Each laboratory sample lot, described above, will receive a unique lot number for data tracking purposes. Lot numbers will be assigned sequentially at the time a laboratory sample lot is established.

Matrix Spike - Positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to accurately measure target concentrations in the absence of undue matrix effects.

Matrix Spike Duplicate - Positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to precisely measure target concentrations in the absence of undue matrix effects.

MB - Method Blank

MBS - Method Blank Spike

MBSD - Method Blank Spike Duplicate

MDL - Method Detection Limit

Method Blank - Negative control prepared in the laboratory to establish that the overall analytical system is not causing significant interference with target analyte detection and quantitation

Method Blank Spike - Positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to accurately measure target concentrations in the absence of undue matrix effects.

Method Blank Spike Duplicate - Positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to precisely measure target concentrations in the absence of undue matrix effects.

Method Detection Limit - Estimate of the lowest level of an analyte that a method can distinguish from noise.

mg/kg – milligrams per kilogram

mg/L – milligrams per liter

mg/m³ - milligrams per cubic meter

MINICAMS[®] - Miniature Continuous Air Monitoring System

MS - Mass Spectroscopy or Matrix Spike

NA - Not Applicable

OSHA - Occupational Safety and Health Act

Performance Evaluation - The analysis of blind samples that are usually part of a study or performance of a group.

Permit - Hazardous Waste Storage Permit

Precision - A measure of an analytical system's agreement between duplicate measurements of the same material. Precision is stated as percent difference (D). When associated with replicate precision determinations on the same material, precision may be stated as mean D and a confidence level.

QAPP - Quality Assurance Program Plan

QA - Quality Assurance

QC - Quality Control

Quality Assurance - The overall system of planning, quality control, and management activities which assure quality.

Quality Control - The specific activities designed to measure quality, including check samples, check sample assessment, audits, reports to management, etc.

Quality Plant (QP) Sample - A quality control (QC) sample used to establish method accuracy and precision. QPs are prepared (in duplicate) in the laboratory by spiking unexposed DAAMS tubes with a solution of dilute chemical agent and, if necessary aspirating with laboratory air to remove residual solvent. QPs are sent into the field with the sample tubes and aspirated with background air.

Quality Control Standard - Used as a calibration check (CC) standard. A standard, prepared at the HL concentration, which verifies the analytical system is operating as designed and is capable of detecting and quantifying chemical agent at the required concentrations.

Quality Laboratory (QL) Standard - A quality control (QC) sample used to verify the initial calibration. QLs are prepared in the laboratory by spiking unexposed DAAMS tubes with a solution of dilute chemical agent and, if necessary, aspirating with laboratory air to remove solvent. QLs are not aspirated with installation air.

R - Range

RCRA - Resource Conservation and Recovery Act

Receiver - During a transfer of custody, the person who is accepting custody of the sample

Recording - Assigning and documenting method numbers and method revision numbers

Relinquisher - During a transfer of custody, the person who is relieved of the sample custody

Reporting Limit - Lowest reportable analyte concentration for a particular sample, usually a factor of 2 to 20 times the MDL

Representativeness - The degree the sample analyzed represents the waste from which it was derived, as measured by field duplicates.

Retention Time Window - A period of time that has been experimentally determined. Peaks eluting during an RTW are tentatively identified as representing the target compound for which the RTW was determined.

Rinse Blank - A sample collected in the field to demonstrate that no cross-contamination has occurred during sampling. For liquid and soil samples, one rinse blank per field sample lot is needed when non-disposable sampling equipment is used. Rinse blanks are not required when sampling equipment is used.

RL - Reporting Limit

RPD - Relative Percent Difference

RSD - Relative Standard Deviation

Sample Collection Lot - Twenty or fewer samples collected from the same waste description at one time (shift) by a single team of sampling personnel.

Sample File - Sample collection information associated with a single sample or a sequential group of samples that share the same sample collection information. The sample file consists of an Analysis Report, Chain-of-Custody/Analysis Request form, Login Checklist, etc.

Sequence - The order of standards and samples in the analytical run

Significant Figures - The number of digits required to express the uncertainty of reported data. The following digits are always significant: 1) the non-zero numbers, 2) zeroes between non-zero numbers, 3) zeroes which are to the right of the decimal point and at the end of the number, and 4) zeroes which are to the left of a written decimal point when the number is ≥ 10 .

Submitting Laboratory - Any laboratory generating labware for submission to the washroom

Support Services Personnel - Personnel responsible for recording and issuing controlled documents

SW-846 - EPA Test Methods for Evaluating Solid Waste

Technical Personnel - Analytical, support, or management personnel responsible for the subject matter of the document

TLV - Threshold Limit Values

UST - Underground Storage Tank

VX - O-ethyl S-(2-diisopropylaminoethyl) methylphosphonothiolate, CAS 50782-69-9, a persistent nerve agent

WAP - Waste Analysis Plan

WDTC - West Desert Test Center